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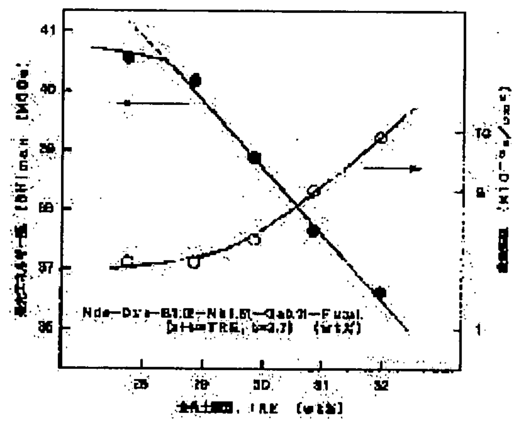
(54) ND-FE-B TYPE SINTERED MAGNET

(57)Abstract:
PURPOSE: To stably provide an Nd-Fe-B type sintered magnet having a large coercive force even without Co and large energy product.
CONSTITUTION: An Nd-Fe-B type sintered magnet comprises 28-32wt.% of R, 0.5-2.0wt.% of B, 0.1-2.0wt.% of Wb, 0.05-1.0wt.% of Ga, 1000-6000ppm of oxygen, and the residue mainly of Fe, wherein its coercive force is 20kOe or more, a maximum magnetic energy product is 30MGOe or more.



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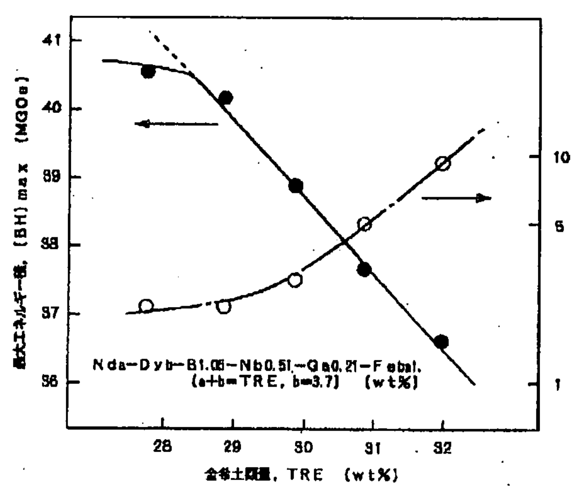
(54)【発明の名称】　Nd－Fe－B型焼結磁石

(57)【要約】

【目的】　Coを含有せずとも保磁力が大きく、かつエネルギー積の大きいNd－Fe－B型焼結磁石を安定的に提供する。

【構成】　28～32wt%のR、0.5～2.0wt%のB、0.1～2.0wt%のNb、0.05～1.0wt%のGa、1000ppm～6000ppmの酸素、残部が主としてFeからなり保磁力が20KOe以上、最大磁気エネルギー積が30MGOe以上であるNd－Fe－B型焼結磁石。



【特許請求の範囲】

【請求項1】 28～32wt%のR（但し、RはY及び希土類元素から選ばれた少なくとも1種であり、Rの内、3．0～8．0wt%がDy，残りは、Nd又はPrの1種又は2種であり、NdをRの内に50at%以上を含む）、0．5～2．0wt%のB、0．1～2．0wt%のNb、0．05～1．0wt%のGa、1000ppm～6000ppmの酸素、及び不可避的不純物を含有し、残部が主としてFeからなり保磁力iHcが20KOe以上、最大磁気エネルギー積（BH）maxが30MGOe以上である耐食性、耐熱性に優れたNd－Fe－B型焼結磁石。

【請求項2】 Ga含有量が0．05～0．8wt%である請求項1記載のNd－Fe－B型焼結磁石。

【請求項3】 Ga含有量が0．1～0．6wt%である請求項1記載のNd－Fe－B型焼結磁石。

【請求項4】 Ga含有量が0．1～0．4wt%である請求項1記載のNd－Fe－B型焼結磁石。

【請求項5】 Rのうち3．0～5．0wt%がDyであり、最大磁気エネルギー積（BH）maxが30MGOe以上である請求項1～請求項4のいずれかに記載のNd－Fe－B型焼結磁石。

【請求項6】 Rのうち5．0～8．0wt%がDyであり、保磁力iHcが25kOe以上である請求項1～請求項4のいずれかに記載のNd－Fe－B型焼結磁石。

【請求項7】 常温磁気特性として

iHc≧20kOe，（BH）max≧30MGOe

であり、23℃から120℃の残留磁束密度Br，保磁力iHc各々の温度係数α，βが

$-0.12 \leqq \alpha \leqq -0.08$%/℃

$-0.65 \leqq \beta \leqq -0.40$%/℃

である請求項1記載のNd－Fe－B型焼結磁石。

【発明の詳細な説明】

【0001】

【産業上の利用分野】本発明はネオジム（Nd）、鉄（Fe）及びホウ素（B）を主成分とする永久磁石に関し、特に優れた耐食性、耐熱性を有するNd－Fe－B型焼結永久磁石に関するものである。

【0002】

【従来の技術】Nd－Fe－B型焼結磁石は、$SmCo_5$型焼結磁石或いはSm_2Co_{17}型焼結磁石と比較して高いエネルギー積（BH）maxを有するので、種々の用途に使用されるようになっている。しかしながら、Nd－Fe－B型焼結磁石は、これらSm－Co型焼結磁石に比較して熱安定性に劣るので、その熱安定性を増す為に種々の試みが提案されている。特開昭64－7503号公報には、熱安定性の良好な永久磁石として一般式：

$R(Fe_{1-x-y-z}Co_xB_yGa_z)_A$

（但し、Rは希土類元素から選ばれた少なくとも1種であり、$0 \leqq x \leqq 0.7$、$0.02 \leqq y \leqq 0.3$、$0.001 \leqq z \leqq 0.15$、$4.0 \leqq A \leqq 7.5$である。）、及び、

$R(Fe_{1-x-y-z}Co_xB_yGa_zM_u)_A$

（但し、Rは希土類元素から選ばれた少なくとも1種であり、MはNb，W，V，Ta及びMoから選ばれた1種または2種以上の元素であり、$0 \leqq x \leqq 0.7$、$0.02 \leqq y \leqq 0.3$、$0.001 \leqq z \leqq 0.15$、$u \leqq 0.1$、$4.0 \leqq A \leqq 7.5$である。）により表されるものを開示している。

【0003】

【発明が解決しようとする課題】しかしながら、Coを含有することなく高耐食性、高耐熱特性を有し、かつ同時に高い水準の保磁力iHc、エネルギー積（BH）maxを兼ね備えたNd－Fe－B型異方性焼結磁石を安定的に生産しようとする場合には前記公知技術を超えて更に詳細な研究・検討に基づいた成分組成範囲の限定、酸化物の限定等が必要であることが分かった。本発明はこのような知見に基づき、特に耐食性、耐熱性に優れたNd－Fe－B型焼結磁石を提供するものである。本発明は、Coを含有せずともDy，Nbを有効に利用することによって著しく耐食性を高め、Dy量、Ga量を特定範囲とすることによって高耐熱性を付与し、同時に希土類量Rを低め、かつ、酸素量を限定することにより保磁力iHcが大きく、かつエネルギー積（BH）maxの大きいNd－Fe－B型焼結磁石を安定的に提供するものである。

【0004】

【課題を解決するための手段】本発明は、28～32wt%のR（但し、RはY及び希土類元素から選ばれた少なくとも1種であり、Rの内、3．0～8．0wt%がDy、残りは、Nd又はPrの1種又は2種であり、NdをRの内に50at%以上を含む）、0．5～2．0wt%のB、0．1～2．0wt%のNb、0．05～1．0wt%のGa、1000ppm～6000ppmの酸素、及び不可避的不純物を含有し、残部が主としてFeからなり保磁力iHcが20KOe以上、最大磁気エネルギー積（BH）maxが30MGOe以上である耐食性、耐熱性に優れたNd－Fe－B型焼結磁石である。本発明の永久磁石の組成の限定理由について、以下詳細に説明する。

【0005】本発明においてRは28～32wt%の範囲で含有される。後述の実施例4に示されるようにR量が32wt%以下と少ないほど（BH）max、および耐食性の向上に有効である。しかし、28wt%未満ではインゴット中にα－Feが発生し易くなり（BH）maxの増大は期待しにくい。よってR量は28～32wt%とする。RはNdを主体とするために、R成分の内、50at%以上のNdを含有するものとする。Rの内には3．0～8．0wt%のDyを含有するが、残部

はNd単独又はNdとPrとの混合である。Prは保磁力iHcの向上に効果がある。

【0006】DyをR成分として3．0～8．0wt%含有することによって、キュリー点Tcが上昇するとともに異方性磁場（H_A）が増大して保磁力iHcが向上し、耐熱性を著しく向上させる。また、Dyは耐食性向上にも効果がある。本発明において、Dyの含有量が3．0wt%より少ないと、熱安定性、耐食性を向上させるという本発明の目的は達成されない。しかし、8．0wt%よりも含有量が多くなると、残留磁束密度Br及び最大エネルギー積（BH）maxの低下による磁気特性の劣化が著しい。したがって、Dyの含有量は3．0～8．0wt%とする。Dyが5．0wt%よりも含有量が多いと、残留磁束密度Br及び最大エネルギー積（BH）maxの低下の低下はあるものの、25kOe以上の保磁力iHcを得ることができる。よってより高保磁力特性を得ようとする場合にはDyの含有量を5．0～8．0wt%とする。逆に大きな残留磁束密度Br及び最大エネルギー積（BH）maxを得ようとする場合にはDyの含有量を3．0～5．0wt%とすればよい。

【0007】Coを含有させるとキュリー点の上昇には効果はあるが、残留磁束密度Br及び保磁力iHcが低下するので、本発明においては不純物以下のレベルとする。Bは0．5wt%未満の場合高保磁力が得られず、一方、2．0wt%を越えると、Bに富んだ非磁性相が増加し、残留磁束密度Brが低下する。そのため0．5～2．0wt%の含有量とする。好ましいBの含有量は0．8～1．2wt%である。

【0008】Gaは、残留磁束密度Brを殆ど低下させず、保磁力iHcを向上する効果がある。Ga含有量が0．05wt%未満の場合は保磁力iHcを向上する効果が十分でない。Ga含有量が1．0wt%を越えると、残留磁束密度Brが低下し、所望の高エネルギー積が得られない。よって、Ga含有量は0．05～1．0wt%とする。Ga含有量が多いと磁石のヒステリシスカーブの角形性が悪くなるので、高い角形性を付与するためにも好ましいGaの含有量は0．05～0．8wt%である。より好ましいGaの含有量は0．1～0．6wt%である。更に好ましくは0．1～0．4wt%である。

【0009】本発明の永久磁石は、上記成分の他に0．1～2．0wt%のNbを含有する。Nbは焼結時に結晶粒が粗大化することを抑制する効果がある。この効果により、保磁力iHcが向上し、ヒステリシスカーブの角型性が良好になる。また、焼結体の結晶粒が微細になることは磁石は良好な着磁性を有することに寄与する。着磁性の良好であり優れた耐熱性を有するNd－Fe－B型焼結磁石を得る。耐熱性を有する磁石にNbは有効な添加物である。Nbの含有量が0．1wt%未満の場合、粗大粒を抑制する効果が不十分である。一方、Nbの含有量が2．0wt%を越える場合には、NbもしくはNb－Feの非磁性ホウ化物が多く発生し、残留磁束密度Br及びキュリー点Tcが著しく低下するので好ましくない。よって、Nbの含有量は0．1～2．0wt%とする。好ましくは、0．1～1．0wt%である。

【0010】酸素含有量は、1000～6000ppmとする。酸素が1000ppmより少ない場合には磁石粉、及びその圧密体が発火しやすく工業生産上危険がある。一方、6000ppmより多い場合には希土類R成分と反応して希土類酸化物を形成し、高保磁力及び高エネルギー積の磁石を得ることが困難になる。本発明の焼結磁石は、次のようにして製造することができる。まず、一定の成分組成を有するインゴットを真空溶解で製作し、次にこのインゴットを粗粉砕することにより粒径500μm程度の粗粉を得る。この粗粉をジェットミルを用い、不活性ガス雰囲気で微粉砕し、平均粒径3．0～6．0μm（F．S．S．S．）の微粉を得る。次に、この微粉を配向磁場15kOe、成形圧力1．5ton／cm^2の条件下で磁場中プレス成形後、1000～1150℃の温度範囲で焼結する。

【0011】焼結後の熱処理は次のように行なうことができる。まず成形体を焼結して得た焼結体をいったん室温まで冷却する。焼結後の冷却速度は最終製品の保磁力iHcに殆ど影響を与えない。次いで、800～1000℃の温度に加熱し、0．2～5時間保持する。これを第1次熱処理とする。加熱温度が800℃未満または1000℃を越える場合、充分な高保磁力が得られない。加熱保持の後で0．3～50℃／分の速度で室温ないし600℃の温度域まで冷却する。冷却速度が50℃／分を越える場合は、時効のために必要な平衡相が得られず、充分な高保磁力が得られない。また、0．3℃／分未満の冷却速度は熱処理に時間を要し、工業生産上経済的でない。好ましくは、0．6～2．0℃／分の冷却速度が選ばれる。冷却終了温度は室温が望ましいが、多少保磁力iHcを犠牲にすれば600℃とし、その温度以下は急冷してもよい。好ましくは、常温～400℃の温度まで冷却する。

【0012】熱処理は更に500～650℃の温度で0．2～3時間行う。これを第2次熱処理とする。組成によって異なるが、好ましくは540～640℃での熱処理が有効である。熱処理温度が500℃未満の場合及び650℃より高い場合は、高保磁力が得られても不可逆減磁率の低下が起こる。熱処理は第1次熱処理と同様、0．3～400℃／分の速度で冷却する。冷却は水中、シリコンオイル中、アルゴン気流中等で行うことができる。冷却速度が400℃／分を越える場合、急冷により焼結体中に亀裂が入り、工業的に価値のある永久磁石材料が得られない。また、冷却速度が0．3℃／分未満の場合、冷却過程で保磁力iHcに好ましくない相が

出現する。

【００１３】

【実施例】以下、実施例により本発明を更に詳細に説明する。

（実施例１）金属Ｎｄ、金属Ｄｙ、Ｆｅ、ｆｅｒｒｏ－Ｂ、ｆｅｒｒｏ－Ｎｂ、金属Ｇａを所定の重量秤量し、これを真空溶解して重量１０ｋｇのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

Ｎｄ27.1－Ｄｙ3.8－Ｂ1.03－Ｎｂ0.58－Ｇａ0.18－Ｆｅbal.（ｗｔ％）

このインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い５００μｍ以下の粒度の粗粉を得た。次いで、この粗粉をジェットミルを用い不活性ガス雰囲気中での微粉砕をして微粉を得た。この微粉は平均粒径４．０μｍ（Ｆ．Ｓ．Ｓ．Ｓ．）であり、含有酸素量が５１００ｐｐｍであった。次に、この微粉を配向磁場強度１５ｋＯｅ、成形圧力１．５ｔｏｎ／ｃｍ^{2}の条件下の横磁場中でプレス成形し、３０ｍｍ×２０ｍｍ×１５ｍｍの成形体を作製した。この成形体は実質的に真空の条件で１０８０℃×３ｈｒの焼結を行い、得られた焼結体に９００℃×２ｈｒの第１次熱処理、次いで５８０℃×２ｈｒの第２次熱処理を施した。得られた焼結体の密度は７．５８ｇ／ｃｍ^3、また含有酸素量は４８００ｐｐｍであった。この試料の常温磁気特性を測定したところ以下の様な値を得た。

Ｂｒ＝１２．７ｋＧ

ｂＨｃ＝１２．０ｋＯｅ

ｉＨｃ＝２２．９ｋＯｅ

（ＢＨ）ｍａｘ＝３８．３ＭＧＯｅ

更にキュリー点Ｔｃとして３１７℃、２３℃から１２０℃のＢｒとｉＨｃの温度係数α、βとして各々－０．１０、－０．５７％／℃の値を得た。またパーミアンス係数Ｐｃ＝１．０，２．０形状をした試料の１００℃での不可逆減磁率は各々１．５，０．８％であり優れた耐熱性を有している。

【００１４】（実施例２）実験条件を変えて、実施例１と同様にして次の実験結果を得た。

組成 ： Ｎｄ25.5－Ｄｙ6.4－Ｂ1.03－Ｎｂ0.55－Ｇａ0.19－Ｆｅbal.（ｗｔ％）

焼結 ： １０８０℃×２ｈｒ

第１次熱処理 ： ９００℃×２ｈｒ

第２次熱処理 ： ５８５℃×２ｈｒ

常温磁気特性 ： Ｂｒ ＝ １２．０３ｋＧ

ｂＨｃ ＝ １１．６ｋＯｅ

ｉＨｃ ＝ ２７．７ｋＯｅ

（ＢＨ）ｍａｘ ＝ ３４．６ＭＧＯｅ

キュリー点 ： Ｔｃ ＝ ３２２℃

不可逆減磁率 ［ａｔ １００℃］ ： Ｐｃ ＝ １．０％ → １．３％

Ｐｃ ＝ ２．０％ → ０．５％

Ｂｒ温度係数（α），ｉＨｃ温度係数（β） ［２３℃～１２０℃］

： α＝ －０．１０％／℃

β＝ －０．５０％／℃

焼結体含有酸素量 ： ４４００ｐｐｍ

実施例１と同様、本実施例も常温磁気特性と共に高温特性にも優れており、耐熱性に優れた磁石を得る事ができる。

【００１５】（実施例３）ジジムメタル（Ｎｄ７０ｗｔ％－Ｐｒ３０ｗｔ％）を使用し、実施例１、２と同様にして次の実験結果を得た。

組成 ： Ｎｄ18.9－Ｐｒ5.1－Ｄｙ7.0－Ｂ1.08－Ｎｂ0.68－Ｇａ0.35－Ｆｅbal.（ｗｔ％）

焼結 ： １０８０℃×２ｈｒ

第１次熱処理 ： ９００℃×２ｈｒ

第２次熱処理 ： ５８０℃×２ｈｒ

常温磁気特性 ： Ｂｒ ＝ １１．９ｋＧ

ｂＨｃ ＝ １１．４ｋＯｅ

ｉＨｃ ＝ ３３．８ｋＯｅ

（ＢＨ）ｍａｘ ＝ ３１．２ＭＧＯｅ

キュリー点 ： Ｔｃ ＝ ３２５℃

不可逆減磁率 ［ａｔ １００℃］ ： Ｐｃ ＝ １．０ → １．２％

Ｐｃ ＝ ２．０ → ０．４％

Ｂｒ温度係数（α），ｉＨｃ温度係数（β） ［２３℃～１２０℃］

： α＝ －０．０９％／℃

β＝ －０．４８％／℃

焼結体含有酸素量 ： ５０００ｐｐｍ

ジジムメタルを用いた場合でも、実施例１、２と同様常温磁気特性、高温特性、耐熱性に優れた磁石を得ることができる。

【００１６】（実施例４）金属Ｎｄ、金属Ｄｙ、Ｆｅ、ｆｅｒｒｏ－Ｂ、ｆｅｒｒｏ－Ｎｂ、金属Ｇａを所定の重量秤量し、これを真空溶解して重量１０ｋｇのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

Ｎｄａ－Ｄｙｂ－Ｂ1.05－Ｎｂ0.51－Ｇａ0.21－Ｆｅbal.
（a+b＝ＴＲＥ， b=3.7） （ｗｔ％）

各々のこのインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い５００μｍ以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径４．０μｍ（Ｆ．Ｓ．Ｓ．Ｓ．）であり、含有酸素量は１０００～９０００ｐｐｍであった。次に、この微粉を配向磁場強度１５ｋＯｅ、成形圧力１．５ｔｏｎ／ｃｍ^{2}の条件下の横磁場中でプレス成形し、３０ｍｍ×２０ｍｍ×１５ｍｍの成形

体を作製した。この成形体は実質的に真空の条件で1080℃×3hrの焼結を行い、得られた焼結体に900℃×2hrの第1次熱処理、次いで580℃×2hrの第2次熱処理を施した。得られた焼結体の密度は7．58～7．62g／cm^3、また含有酸素量は1000～4000ppmであった。この試料について、TRE含有量に対して最大エネルギー積（BH）max及び腐食減量がどのように変化するかを測定し、図5に示すような結果を得た。腐食減量は磁石を温度120℃，湿度90%，気圧1．0atmの環境中に100時間暴露したときに得られたものである。図1に示されるようにTRE量を少なくすることによって（BH）maxを大きくすることが出来るが、28wt%未満とするとインゴット中にα－Feが発生し易くなり（BH）maxの向上は期待しにくい。腐食減量もやはりTRE量を少なくすることにより低減させることができる。これは、TRE量を少なくすることによって腐食しやすいNd－rich相が減少する為である。しかしながら、TRE量を28～32wt%という低い値としても含有酸素量が6000ppmを超えると保磁力iHcの減少が著しくなるため、酸素量は1000～6000ppmとする。図2に焼結体中の酸素含有量と磁気特性の関係を示す。

【0017】（実施例5）金属Nd、金属Dy、Fe、ferro－B、ferro－Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

Nd(31.0-a)－Dya－B1.05－Nb0.59－Gab－Febal.（2.8≦a≦8.5,　0≦b≦1.2）　（wt%）

各々のこのインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い500μm以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径4．0μm（F．S．S．S．）であり、含有酸素量は5500ppmであった。次に、この微粉を配向磁場強度15kOe、成形圧力1．5ton／cm^{2}の条件下の磁場中でプレス成形し、30mm×20mm×15mmの成形体を作製した。この成形体は実質的に真空の条件で1080℃×2．5hrの焼結を行い、得られた焼結体に890℃×2hrの第1次熱処理、次いで580℃×2hrの第2次熱処理を施した。得られた焼結体の密度は7．57～7．63g／cm^3、また含有酸素量は4400～5500ppmであった。これら試料について、常温磁気特性を測定し、図3、図4及び図5に示すような結果を得た。図3はDy＝5．3wt%として測定した結果であるが、Ga含有量が0．05wt%未満では効果を発揮しにくく、また、含有量を1．0wt%以上にしても（BH）maxの低下が著しくなるだけでiHcの向上はさほど期待できないので0．05～1．0wt%が適

量である。GaはDyに比較して（BH）maxを著しく低下することなくiHcを増加させる効果が大きいので0．01～1．0wt%の含有は本発明において必須となる。図4にはGa含有量を0．20wt%としてDy含有量を変化させた結果を示す。Dy含有量の増加はiHcの向上に大きく貢献するが、一方で（BH）maxを著しく低下させるので含有量は3．6～8．0wt%が適量である。図5にはDy含有量をパラメータとして、Ga含有量を0～0．6wt%まで変化させた場合の結果を示す。Dy含有量が8．0wt%を越えると（BH）maxが著しく低下する。また、Dy含有量が3．0wt%未満であると、20kOeを越える高保磁力が得にくい。

【0018】（実施例6）ジジムメタル（Nd70wt%－Pr30wt%）、金属Dy、Fe、ferro－B、ferro－Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

（Nd＋Pr）28.1－Dy3.6－B1.03－Nb0.53－Gab－Febal.　（0≦b≦0.6）　（wt%）

各々のこのインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い500μm以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をし微粉を得た。この微粉は平均粒径3．5μm（F．S．S．S．）であり、含有酸素量は4800ppmであった。次に、この微粉を配向磁場強度15kOe、成形圧力1．5ton／cm^{2}の条件下の横磁場中でプレス成形し、30mm×20mm×15mmの成形体を作製した。この成形体は実質的に真空の条件で1080℃×3hrの焼結を行い、得られた焼結体に890℃×2hrの第1次熱処理、次いで580℃×2hrの第2次熱処理を施した。得られた焼結体の密度は7．60g／cm^3、また含有酸素量は1800～2400ppmであった。これら試料について、常温磁気特性を測定し、図6に示すような結果を得た。図6に示されるようにGaを含有させることによって保磁力iHc及びHkの向上が認められるので0．05wt%以上の含有が必須となる。しかしながら、Gaが0．4wt%を超えるとHkが低下しヒステリシスループの角形性が低下するので上限は1．0wt%ではあるが、好ましくは0．8wt%、より好ましくは0．6wt%、更に好ましくは0．4wt%である。

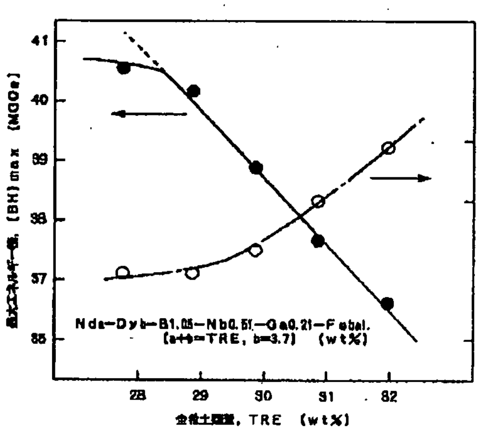
【0019】（実施例7）ジジムメタル（Nd70wt%－Pr30wt%）、金属Dy、Fe、ferro－B、ferro－Nb、金属Gaを所定の重量秤量し、これを真空溶解して重量10kgのインゴットを作製した。このインゴットの成分分析を行なうと重量比で以下のような組成であった。

（Ｎｄ＋Ｐｒ）27.0－Ｄｙ5.0－Ｂ1.03－Ｎｂx－Ｇａ0.15－Ｆｅbal.（0≦x≦1.0）　（ｗｔ％）
各々のこのインゴットをハンマーで解砕した後、さらに粗粉砕機を用い不活性ガス雰囲気中での粗粉砕を行い５００μｍ以下の粒度の粗粉を得た。この粗粉を同じくジェットミルを用い不活性ガス雰囲気中で微粉砕をして微粉を得た。この微粉は平均粒径３．５μｍ（Ｆ．Ｓ．Ｓ．Ｓ．）であり、含有酸素量は４９００ｐｐｍであった。次に、この微粉を配向磁場強度１５ｋＯｅ、成形圧力１．５ｔｏｎ／ｃｍ^{2}の条件下の磁場中でプレス成形し、３０ｍｍ×２０ｍｍ×１５ｍｍの成形体を作製した。この成形体は実質的に真空の条件で１０８０℃×３ｈｒの焼結を行い、得られた焼結体に８９０℃×２ｈｒの第１次熱処理、次いで５８０℃×２ｈｒの第２次熱処理を施した。得られた焼結体の密度は７．５９～７．６３ｇ／ｃｍ^3、また含有酸素量は４４００～５３００ｐｐｍであった。これら試料について、常温磁気特性、および平均粒径を測定し、図７に示すような結果を得た。図７に示されるようにＮｂを含有させることにより焼結時の結晶粒成長を抑制でき、その結果焼結体平均粒径を小さくすることができる。そして、この効果により保磁力ｉＨｃの向上が期待できる。２．０ｗｔ％以上の含有によってでは結晶粒成長の抑制効果はさほど期待できず、最大エネルギー積（ＢＨ）ｍａｘの低下も大きくなるので０．４～２．０ｗｔ％の含有が適量である。

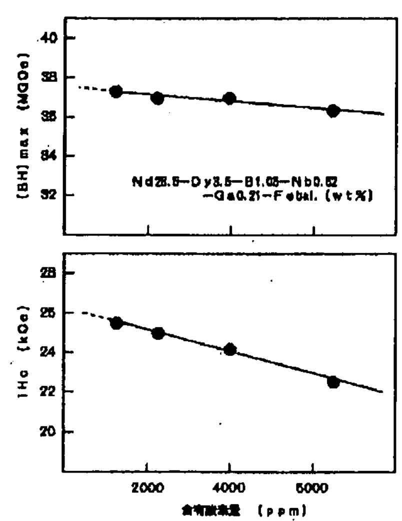
【００２０】
【発明の効果】以上、実施例にも示したようにＮｄ－Ｆｅ－Ｂ型焼結磁石にＧａ、Ｎｂを複合添加し、Ｄｙ量、酸素量などを適切なものとして高保磁力かつ高エネルギー積を有し高耐熱性、高耐食性の磁石を得る。

【図面の簡単な説明】
【図１】Ｎｄ－Ｆｅ－Ｂ型焼結磁石の全希土類量に対する最大エネルギー積（ＢＨ）ｍａｘ、腐食減量の変化を示したグラフ。
【図２】Ｎｄ－Ｆｅ－Ｂ型焼結磁石の含有酸素量に対する最大エネルギー積（ＢＨ）ｍａｘ、保磁力ｉＨｃ、腐食減量の変化を示したグラフ。
【図３】Ｎｄ－Ｆｅ－Ｂ型焼結磁石のＧａ含有量（０～１．２ｗｔ％）に対する最大エネルギー積（ＢＨ）ｍａｘ、保磁力ｉＨｃの変化を示したグラフ。
【図４】Ｎｄ－Ｆｅ－Ｂ型焼結磁石のＧａ含有量に対する最大エネルギー積（ＢＨ）ｍａｘ、保磁力ｉＨｃ、腐食減量の変化を示したグラフ。
【図５】Ｎｄ－Ｆｅ－Ｂ型焼結磁石のＧａ含有量（０～０．６ｗｔ％）及びＤｙ含有量に対する最大エネルギー積（ＢＨ）ｍａｘ、保磁力ｉＨｃの変化を示したグラフ。
【図６】Ｎｄ－Ｆｅ－Ｂ型焼結磁石のＧａ含有量（０～０．６ｗｔ％）に対する最大エネルギー積（ＢＨ）ｍａｘ、保磁力ｉＨｃ、角型性の変化を示したグラフ。
【図７】Ｎｄ－Ｆｅ－Ｂ型焼結磁石のＮｂ含有量に対する焼結体平均粒径、最大エネルギー積（ＢＨ）ｍａｘの変化を示したグラフ。

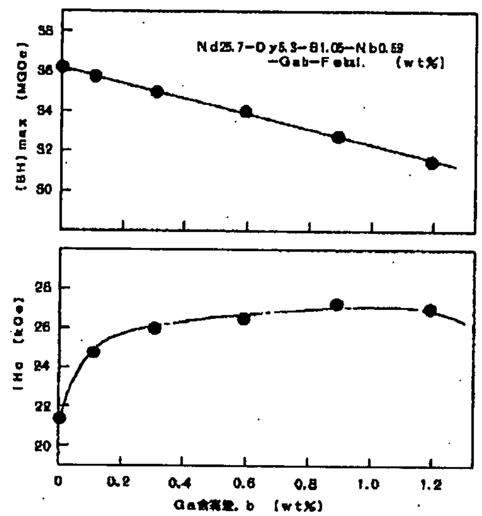
【図１】



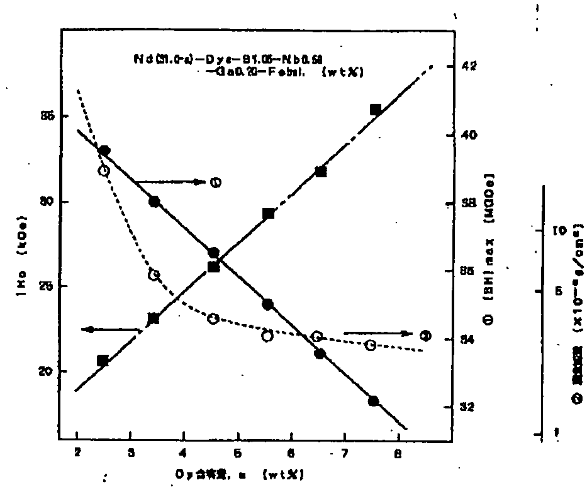
【図２】



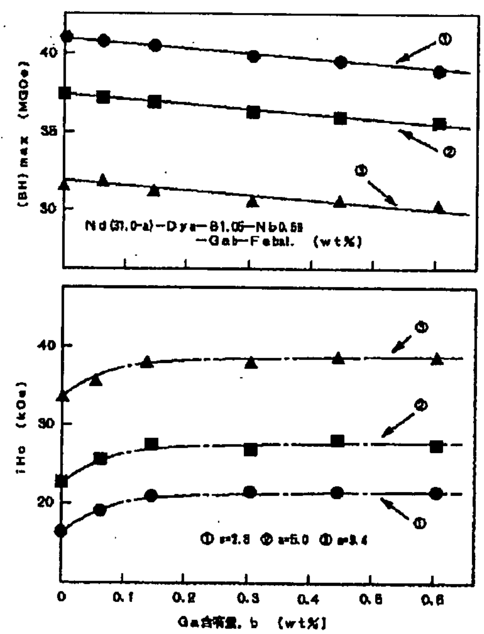
【図3】


Nd25.7−Dy5.3−B1.05−Nb0.59
−Gab−Febal. (wt%)
(BH)max (MGOe)
iHc (kOe)
Ga含有量, b (wt%)

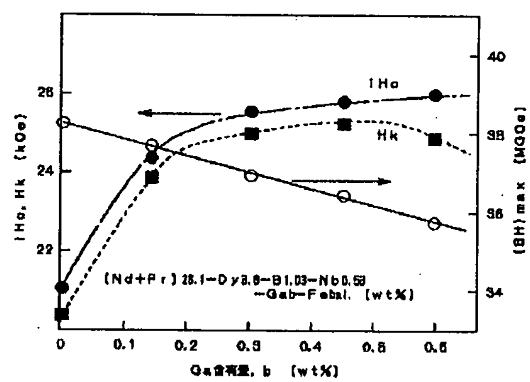
【図4】


Nd(31.0-a)−Dya−B1.05−Nb0.59
−Ga0.20−Febal. (wt%)
iHc (kOe)
① (BH)max (MGOe)
Dy含有量, a (wt%)

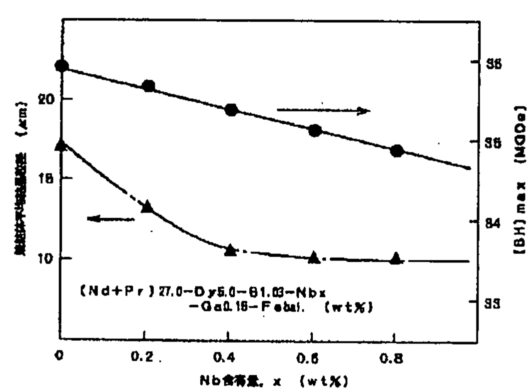
【図5】


Nd(31.0-a)−Dya−B1.05−Nb0.59
−Gab−Febal. (wt%)
(BH)max (MGOe)
iHc (kOe)
Ga含有量, b (wt%)

【図6】


iHc
Hk
(Nd+Pr)28.1−Dy3.6−B1.03−Nb0.53
−Gab−Febal. (wt%)
iHc, Hk (kOe)
(BH)max (MGOe)
Ga含有量, b (wt%)

【図7】


(Nd+Pr)27.0−Dy5.0−B1.03−Nbx
−Ga0.15−Febal. (wt%)
(BH)max (MGOe)
Nb含有量, x (wt%)

Bibliography

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H01F 1/053
[FI]
H01F 1/04 H
[Request for Examination] Un-asking.
[The number of claims] 7
[Mode of Application] OL
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[Translation done.]

Epitome

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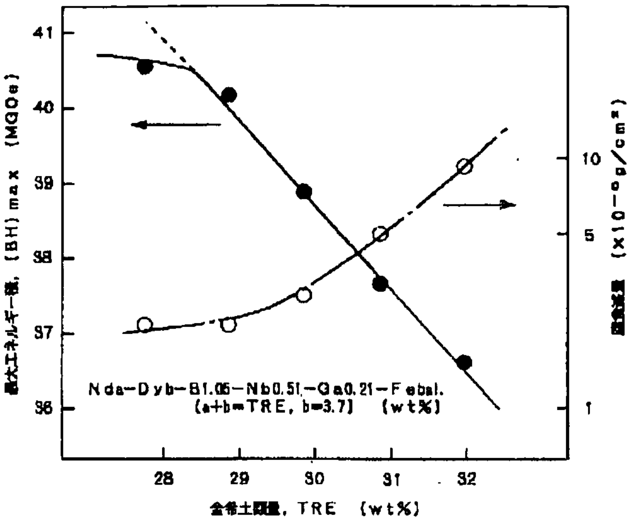
(57) [Abstract]
[Objects of the Invention] Co is not contained but the large Nd-Fe-B mold sintered magnet of an energy product is offered stably greatly [** / coercive force].
[Elements of the Invention] A 28 - 32wt% R, a 0.5 - 2.0wt% B, a 0.1 - 2.0wt% Nb, a 0.05 - 1.0wt% Ga, 1000 ppm - 6000 ppm oxygen, the Nd-Fe-B mold sintered magnet 20 or more KOes and whose maximum magnetic energy product the remainder mainly consists of Fe and coercive force is 30 or more MGOes.

[Translation done.]



[Translation done.]

* NOTICES *

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1. This document has been translated by computer. So the translation may not reflect the original precisely.
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

CLAIMS

[Claim(s)]
[Claim 1] 28 - 32wt% R (however, R is at least one sort chosen from Y and rare earth elements 3.0 - 8.0wt% among R Dy and the remainder) Are one sort of Nd or Pr, or two sorts, and more than 50at% is included for Nd in the inside of R. A 0.5 - 2.0wt% B, a 0.1 - 2.0wt% Nb, 0.05 - 1.0wt% Ga, The Nd-Fe-B mold sintered magnet which contained 1000 ppm - 6000 ppm oxygen and an unescapable impurity, and the remainder mainly consisted of Fe, and was excellent in the corrosion resistance whose coercive force iHc is 20 or more KOes, and whose maximum magnetic energy (product BH) max is 30 or more MGOes, and thermal resistance.
[Claim 2] The Nd-Fe-B mold sintered magnet according to claim 1 whose Ga content is 0.05 - 0.8wt%.

[Claim 3] The Nd-Fe-B mold sintered magnet according to claim 1 whose Ga content is 0.1 - 0.6wt%.
[Claim 4] The Nd-Fe-B mold sintered magnet according to claim 1 whose Ga content is 0.1 - 0.4wt%.
[Claim 5] The Nd-Fe-B mold sintered magnet according to claim 1 to 4 whose 3.0 - 5.0wt% is Dy among R and whose maximum magnetic energy (product BH) max is 30 or more MGOes.
[Claim 6] The Nd-Fe-B mold sintered magnet according to claim 1 to 4 whose 5.0 - 8.0wt% is Dy among R and whose coercive force iHc is 25 or more kOes.
[Claim 7] as ordinary temperature magnetic properties -- iHc>=20kOe and (BH) max>=30MGOe -- it is -- the residual magnetic flux density Br of 23 to 120 degrees C, and coercive force iHc -- the Nd-Fe-B mold sintered magnet according to claim 1 each temperature coefficients alpha and beta of whose are -0.12 <=alpha<=-0.08%/degree-C-0.65 <=beta<=-0.40%/degrees C.

[Translation done.]

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Industrial Application] This invention relates to the Nd-Fe-B mold sintering permanent magnet which has the especially excellent corrosion resistance and thermal resistance about the permanent magnet which uses neodymium (Nd), iron (Fe), and boron (B) as a principal component.
[0002]
[Description of the Prior Art] Since a Nd-Fe-B mold sintered magnet has high energy product (BH) max as compared with a SmCo5 mold sintered magnet or an Sm2Co17 mold sintered magnet, it is used for various

applications. However, since a Nd-Fe-B mold sintered magnet is inferior to thermal stability as compared with these Sm-Co mold sintered magnet, and the thermal stability is increased, various attempts are proposed. In JP,64-7503,A, as a good permanent magnet of thermal stability, general formula:R(Fe1-x-y-zCoxByGaz) A (However, R is at least one sort chosen from rare earth elements, and is 0<=x<=0.7, 0.02<=y<=0.3, 0.001<=z<=0.15, and 4.0<=A<=7.5.) And R(Fe1-x-y-zCoxByGazMu) A (However, R is at least one sort chosen from rare earth elements, and M is one sort or two sorts or more of elements chosen from Nb, W, V, Ta, and Mo, and is 0<=x<=0.7, 0.02<=y<=0.3, 0.001<=z<=0.15, u<=0.1, and 4.0<=A<=7.5.) What is expressed is indicated.

[0003]

[Problem(s) to be Solved by the Invention] However, when it was going to produce stably the Nd-Fe-B mold anisotropy sintered magnet which has high corrosion resistance and a high heatproof property, and has the coercive force iHc of a high level, and energy product (BH) max in coincidence, without containing Co, it turned out exceeding said well-known technique that it limits [the limitation of the component presentation range based on still more detailed research and examination, / of an oxide]. Especially this invention offers the Nd-Fe-B mold sintered magnet excellent in corrosion resistance and thermal resistance based on such knowledge. When this invention does not contain Co but ** also uses Dy and Nb effectively, coercive force iHc offers stably the large Nd-Fe-B mold sintered magnet of energy product (BH) max greatly by raising corrosion resistance remarkably, giving high thermal resistance by making the amount of D(ies), and the amount of Ga(s) into the specific range, and lowering the amount R of rare earth to coincidence, and limiting the amount of oxygen.

[0004]

[Means for Solving the Problem] This invention is 28 - 32wt% R (however, R is at least one sort chosen from Y and rare earth elements 3.0 - 8.0wt% among R Dy and the remainder). Are one sort of Nd or Pr, or two sorts, and more than 50at% is included for Nd in the inside of R. A 0.5 - 2.0wt% B, a 0.1 - 2.0wt% Nb, 0.05 - 1.0wt% Ga, It is the Nd-Fe-B mold sintered magnet which contained 1000 ppm - 6000 ppm oxygen and an unescapable impurity, and the remainder mainly consisted of Fe, and was excellent in the corrosion resistance whose coercive force iHc is 20 or more KOes, and whose maximum magnetic energy (product BH) max is 30 or more MGOes, and thermal resistance. The reason for limitation of a presentation of the permanent magnet of this invention is explained to a detail below.

[0005] In this invention, R is contained in 28 - 32wt%. It is so effective in (BH) max and corrosion resistance improvement that there are few amounts of R as less than [32wt%] as shown in the below-mentioned example 4. However, less than [28wt%], it becomes easy to generate alpha-Fe in an ingot, and is hard to expect increase of (BH) max. Therefore, the amount of R is made into 28 - 32wt%. R shall contain Nd beyond 50at% among R components, in order to make Nd into a subject. Although 3.0 - 8.0wt% Dy is contained in the inside of R, the remainder is mixing with a Nd independent, or Nd and Pr. Pr has effectiveness in improvement in coercive force iHc.
[0006] Dy -- R component -- carrying out -- 3.0 - 8.0wt% -- by containing, while Curie point Tc goes up, an anisotropy magnetic field (HA) increases, coercive force iHc improves, and thermal resistance is raised remarkably. Moreover, Dy has effectiveness also in corrosion-resistant improvement. In this invention, if there are few contents of Dy than 3.0wt(s)%, the purpose of this invention of raising thermal stability and corrosion resistance will not be attained. However, when a content increases more than 8.0wt%, degradation of a residual magnetic flux density Br and the magnetic properties by the fall of maximum energy product (BH) max is remarkable. Therefore, the content of Dy is made into 3.0 - 8.0wt%. If a content has more Dy than 5.0wt(s)%, the fall of a fall of a residual magnetic flux density Br and maximum energy product (BH) max can acquire the coercive force iHc of 25 or more kOes of a certain thing. Therefore, the content of Dy is made into 5.0 - 8.0wt% when it is going to acquire a high coercive force property more. Conversely, what is necessary is just to make the content of Dy into 3.0 - 5.0wt%, when it is going to obtain a big residual magnetic flux density Br and big maximum energy product (BH) max.
[0007] When Co is made to contain, there is effectiveness in the rise of the Curie point, but since a residual magnetic flux density Br and coercive force iHc decline, in this invention, it considers as the level below an impurity. If B exceeds 2.0wt% on the other hand by not acquiring quantity coercive force in the case of below 0.5wt%, the nonmagnetic phase which was rich in B will increase, and a residual magnetic flux density Br will fall. Therefore, it considers as a 0.5 - 2.0wt% content. The content of desirable B is 0.8 - 1.2wt%.
[0008] Ga does not almost reduce a residual magnetic flux density Br, and is effective in improving coercive force iHc. When Ga content is less than [0.05wt%], the effectiveness of improving coercive force iHc is not enough. If Ga content exceeds 1.0wt(s)%, a residual magnetic flux density Br will fall and a desired high energy product will not be

obtained. Therefore, Ga content is made into 0.05 - 1.0wt%. Since the square shape nature of a magnetic hysterics TERISU curve will worsen if there are many Ga contents, also in order to give high square shape nature, the desirable content of Ga is 0.05 - 0.8wt%. The more desirable content of Ga is 0.1 - 0.6wt%. Furthermore, it is 0.1 - 0.4wt% preferably.

[0009] The permanent magnet of this invention contains 0.1 - 2.0wt% Nb other than the above-mentioned component. Nb has the effectiveness which controls that crystal grain makes it big and rough at the time of sintering. According to this effectiveness, coercive force iHc improves and the square shape nature of a hysteresis curve becomes good. Moreover, that the crystal grain of a sintered compact becomes detailed contributes a magnet to having good magnetization nature. The Nd-Fe-B mold sintered magnet which has the thermal resistance which is the fitness of magnetization nature and was excellent is obtained. Nb is an effective additive at the magnet which has thermal resistance. When the content of Nb is less than [0.1wt%], the effectiveness which controls a big and rough grain is inadequate. On the other hand, since many nonmagnetic borides of Nb or Nb-Fe occur and a residual magnetic flux density Br and Curie point Tc fall remarkably when the content of Nb exceeds 2.0wt(s)%, it is not desirable. Therefore, the content of Nb is made into 0.1 - 2.0wt%. Preferably, it is 0.1 - 1.0wt%.

[0010] An oxygen content is set to 1000-6000 ppm. When there is less oxygen than 1000 ppm, there is industrial production top risk that magnet powder and its consolidation object tend to ignite. On the other hand, in [than 6000 ppm] more, it reacts with a rare earth R component, a rare earth oxide is formed, and it becomes difficult to obtain the magnet of high coercive force and a high energy product. The sintered magnet of this invention can be manufactured as follows. First, the ingot which has a fixed component presentation is manufactured by vacuum melting, and coarse powder with a particle size of about 500 micrometers is obtained by next carrying out coarse grinding of this ingot. This coarse powder is pulverized in an inert gas ambient atmosphere using a jet mill, and fines with a mean particle diameter of 3.0-6.0 micrometers (F. S.S.S.) are obtained. Next, these fines are sintered in a 1000-1150-degree C temperature requirement after press forming in a magnetic field under the conditions of orientation magnetic field 15kOe and compacting pressure 1.5 ton/cm2.

[0011] Heat treatment after sintering can be performed as follows. The sintered compact which sintered and acquired the Plastic solid first is once cooled to a room temperature. The cooling rate after sintering

hardly affects the coercive force iHc of a final product. Subsequently, it heats in temperature of 800-1000 degrees C, and holds for 0.2 to 5 hours. This is considered as the first heat treatment. When heating temperature exceeds less than 800 degrees C or 1000 degrees C, sufficient high coercive force is not acquired. It cools to a room temperature thru/or a 600-degree C temperature region the rate for 0.3-50-degree-C/after heating maintenance. When a cooling rate exceeds a part for 50-degree-C/, a balanced phase required for aging is not obtained and sufficient high coercive force is not acquired. Moreover, by 0.3-degree-C/, heat treatment takes time amount to the cooling rate of the following, and it is not economical on industrial production. Preferably, 0.6-2.0-degree-C cooling rate for /is chosen. Although cooling termination temperature has a desirable room temperature, as long as it sacrifices coercive force iHc for some, it may consider as 600 degrees C and below the temperature may quench. Preferably, it cools to ordinary temperature -400 degree C temperature.
[0012] Heat treatment is performed at the temperature of further 500-650 degrees C for 0.2 to 3 hours. This is considered as the second heat treatment. Although it changes with presentations, heat treatment at 540-640 degrees C is preferably effective. When heat treatment temperature is less than 500 degrees C, and when higher than 650 degrees C, even if high coercive force is acquired, decline in an irreversible demagnetizing factor takes place. Heat treatment is cooled the rate for 0.3-400-degree-C/like the first heat treatment. Cooling can be performed by the argon air-current middle class underwater and among a silicone oil. When a cooling rate exceeds a part for 400-degree-C/, a crack enters into a sintered compact by quenching, and the permanent magnet ingredient which is industrially worthy is not obtained. Moreover, when a cooling rate is the following by 0.3-degree-C/, the phase which is not desirable appears in coercive force iHc in a cooling process.
[0013]
[Example] Hereafter, an example explains this invention to a detail further.
(Example 1) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy and Fe, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.
Nd27.1-Dy3.8-B1.03-Nb0.58-Ga0.18-Febal. (wt%)
After cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the

coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Subsequently, pulverizing in an inert gas ambient atmosphere was carried out for this coarse powder using the jet mill, and fines were obtained. These fines were 4.0 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 5100 ppm. Next, press forming of these fines was carried out all over the horizontal magnetic field under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm2, and the 30mmx20mmx15mm Plastic solid was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.58 g/cm3 and the amount of content oxygen of the consistency of the obtained sintered compact were 4800 ppm. The following values were acquired when the ordinary temperature magnetic properties of this sample were measured.
Br -- =12.7kGbHc=12.0kOeiHc=22.9kOe(BH) max= -- each -0.10 and a -0.57%/degree C value were further acquired as Curie point Tc 38.3 MGOe as temperature coefficients alpha and beta of Br and iHc of 23 to 120 degrees C. [317 degrees C and] Moreover, the 100-degree C irreversible demagnetizing factor of the sample which carried out permeance coefficient Pc=1.0 and 2.0 configuration has the thermal resistance which is 1.5 and 0.8% respectively, and was excellent.
[0014] (Example 2) Experiment conditions were changed and the following experimental result was obtained like the example 1.
Presentation : Nd25.5-Dy6.4-B1.03-Nb0.55-Ga0.19-Febal. (wt%)
Sintering : The 1080 degree-Cx2hr first heat treatment : The 900 degree-Cx2hr second heat treatment : 585 degree-Cx2hr ordinary temperature magnetic properties : Br = 12.03kGbHc = 11.6kOeiHc = 27.7kOe(BH) max = 34.6MGOe Curie point : Tc = 322-degree-C irreversible demagnetizing factor [at 100 degree C] : Pc = 1.0% -> 1.3%Pc = 2.0% -> 0.5%Br temperature coefficient (alpha), iHc temperature coefficient (beta) [23 degrees C - 120 degrees C]
: alpha= -0.10%/degree-Cbeta= The amount of -0.50% [/degree C] sintered compact content oxygen : Like the 4400 ppm example 1, this example is also excellent also in the elevated-temperature property with ordinary temperature magnetic properties, and the magnet excellent in thermal resistance can be obtained.
[0015] (Example 3) Didym metal (Nd70wt%-Pr30wt%) was used and the following experimental result was obtained like examples 1 and 2.
Presentation : Nd18.9-Pr5.1-Dy7.0-B1.08-Nb0.68-Ga0.35-Febal. (wt%)

sintering : The 1080 degree-Cx2hr first heat treatment : The 900 degree-Cx2hr second heat treatment : 580-degree-Cx2hr ordinary temperature magnetic properties : Br = 11.9kGbHc = 11.4kOeiHc = 33.8kOe(BH) max = 31.2MGOe Curie point : Tc = 325-degree-C irreversible demagnetizing factor [at 100 degree C] : Pc = 1.0 -> 1.2%Pc = 2.0 -> 0.4%Br temperature coefficient (alpha), iHc temperature coefficient (beta) [23 degrees C - 120 degrees C]
: alpha= -0.09%/degree-Cbeta= The amount of -0.48% [/degree C] sintered compact content oxygen : Even when 5000 ppm didym metal is used, the magnet which was excellent in ordinary temperature magnetic properties, an elevated-temperature property, and thermal resistance like examples 1 and 2 can be obtained.
[0016] (Example 4) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy and Fe, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.
Nda-Dyb-B1.05-Nb0.51-Ga0.21-Febal.(a+b=TRE, b=3.7) (wt%
In each, after cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 4.0 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 1000-9000 ppm. Next, press forming of these fines was carried out all over the horizontal magnetic field under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm2, and the 30mmx20mmx15mm Plastic solid was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 900 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.58 - 7.62 g/cm3 and the amount of content oxygen of the consistency of the obtained sintered compact were 1000-4000 ppm. The result as measures how maximum energy product (BH) max and corrosion weight loss change to a TRE content about this sample and shows it to drawing 5 was obtained. Corrosion weight loss is obtained when a magnet is exposed for 100 hours into the temperature of 120 degrees C, 90% of humidity, and the environment of atmospheric-pressure 1.0atm. As shown in drawing 1 , (BH) max can be enlarged by lessening the amount of TRE(s), but if it is less than [28wt%], it becomes easy to generate

alpha-Fe in an ingot, and is hard to expect the improvement in (BH) max. It can be made to decrease by lessening the amount of corrosion-weight-loss mist beam TRE(s). This is for the Nd-rich phase which is easy to corrode by lessening the amount of TRE(s) to decrease. However, since reduction of coercive force iHc will become remarkable if the amount of content oxygen exceeds 6000 ppm also as a low value of 28 - 32wt% for the amount of TRE(s), the amount of oxygen is set to 1000-6000 ppm. The oxygen content in a sintered compact and the relation of magnetic properties are shown in drawing 2 .

[0017] (Example 5) Predetermined carried out weight weighing capacity of Metal Nd, Metals Dy and Fe, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

Nd(31.0-a)-Dya-B1.05-Nb0.59-Gab-Febal. (2.8<=a<=8.5, 0 <=b<=1.2) (wt%)

In each, after cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 4.0 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 5500 ppm. Next, press forming of these fines was carried out all over the magnetic field under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm2, and the 30mmx20mmx15mm Plastic solid was produced. This Plastic solid sintered 1080 degree-Cx2.5hr on condition that the vacuum substantially, was first-heat-treated [of 890 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.57 - 7.63 g/cm3 and the amount of content oxygen of the consistency of the obtained sintered compact were 4400-5500 ppm. About these samples, ordinary temperature magnetic properties were measured and the result as shown in drawing 3 , drawing 4 , and drawing 5 was obtained. Although drawing 3 is the result of measuring as Dy=5.3wt%, since the improvement in iHc is not so much expectable only by Ga content being unable to demonstrate effectiveness easily less than [0.05wt%], and the fall of (BH) max becoming remarkable as for more than 1.0wt% about a content, 0.05 - 1.0wt% is optimum dose. Since the effectiveness of making iHc increasing is large, without Ga falling (BH) max remarkably as compared with Dy, 0.01 - 1.0wt% of content becomes indispensable in this invention. The result to which Dy content was changed, having used Ga

content as 0.20wt(s)% at drawing 4 is shown. Although the increment in Dy content contributes to improvement in iHc greatly, since (BH) max is remarkably reduced by one side, 3.6 - 8.0wt% of a content is optimum dose. The result at the time of changing Ga content to 0 - 0.6wt% is shown by making Dy content into a parameter at drawing 5 . If Dy content exceeds 8.0wt(s)%, (BH) max will fall remarkably. Moreover, it is hard to acquire the high coercive force which exceeds 20kOe as Dy content is less than [3.0wt%].

[0018] (Example 6) Predetermined carried out weight weighing capacity of didym metal (Nd70wt%-Pr30wt%), Metals Dy and Fe, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio.

(Nd+Pr) 28.1-Dy3.6-B1.03-Nb0.53-Gab-Febal. (0<=b<=0.6) (wt%)

In each, after cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.5 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 4800 ppm. Next, press forming of these fines was carried out all over the horizontal magnetic field under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm2, and the 30mmx20mmx15mm Plastic solid was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 890 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.60 g/cm3 and the amount of content oxygen of the consistency of the obtained sintered compact were 1800-2400 ppm. About these samples, ordinary temperature magnetic properties were measured and the result as shown in drawing 6 was obtained. Since improvement in coercive force iHc and Hk is accepted by making Ga contain as shown in drawing 6 , content beyond 0.05wt% becomes indispensable. however -- although an upper limit is 1.0wt(s)% since Hk will fall and the square shape nature of the hysteresis loop will fall, if Ga exceeds 0.4wt(s)% -- desirable -- 0.8wt(s)% -- more -- desirable -- 0.6wt(s)% -- it is 0.4wt(s)% still more preferably.

[0019] (Example 7) Predetermined carried out weight weighing capacity of didym metal (Nd70wt%-Pr30wt%), Metals Dy and Fe, ferro-B, ferro-Nb, and the metal Ga, vacuum melting of this was carried out, and the ingot with

a weight of 10kg was produced. When component analysis of this ingot was performed, they were the following presentations in the weight ratio. (Nd+Pr) 27.0-Dy5.0-B1.03-Nbx-Ga0.15-Febal. (0<=x<=1.0) (wt%)
In each, after cracking this ingot with a hammer, coarse grinding in the inside of an inert gas ambient atmosphere was further performed using the coarse crusher, and coarse powder with a grain size of 500 micrometers or less was obtained. Similarly this coarse powder was pulverized in the inert gas ambient atmosphere using the jet mill, and fines were obtained. These fines were 3.5 micrometers (F. S.S.S.) in mean particle diameter, and the amount of content oxygen was 4900 ppm. Next, press forming of these fines was carried out all over the magnetic field under the conditions of orientation magnetic-field-strength 15kOe and compacting pressure 1.5 ton/cm2, and the 30mmx20mmx15mm Plastic solid was produced. This Plastic solid sintered 1080 degree-Cx3hr on condition that the vacuum substantially, was first-heat-treated [of 890 degree-Cx2hr] to the obtained sintered compact, ranked second to it, and performed the second heat treatment of 580 degree-Cx2hr. 7.59 - 7.63 g/cm3 and the amount of content oxygen of the consistency of the obtained sintered compact were 4400-5300 ppm. About these samples, ordinary temperature magnetic properties and mean particle diameter were measured, and the result as shown in drawing 7 was obtained. By making Nb contain, as shown in drawing 7 , the grain growth at the time of sintering can be controlled, and, as a result, sintered compact mean particle diameter can be made small. And improvement in coercive force iHc is expectable with this effectiveness. 2. Since the depressor effect of grain growth cannot be then expected so much but the fall of maximum energy product (BH) max also becomes large by content beyond 0wt%, 0.4 - 2.0wt% of content is optimum dose.
[0020]
[Effect of the Invention] As mentioned above, as shown also in the example, compound addition of Ga and the Nb is carried out at a Nd-Fe-B mold sintered magnet, and it has high coercive force and a high energy product for the amount of D(ies), the amount of oxygen, etc. as a suitable thing, and high thermal resistance and the magnet of high corrosion resistance are obtained.

[Translation done.]

* NOTICES *

DESCRIPTION OF DRAWINGS

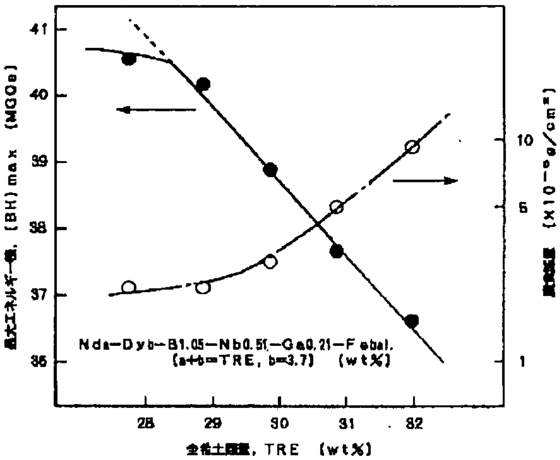
[Brief Description of the Drawings]
[Drawing 1] Maximum energy product (BH) max to the total amount of rare earth of a Nd-Fe-B mold sintered magnet, the graph which showed change of corrosion weight loss.
[Drawing 2] The graph which showed change of maximum energy product (BH) max to the amount of content oxygen of a Nd-Fe-B mold sintered magnet, coercive force iHc, and corrosion weight loss.
[Drawing 3] Maximum energy product (BH) max to Ga content (0 - 1.2wt%) of a Nd-Fe-B mold sintered magnet, the graph which showed change of coercive force iHc.
[Drawing 4] The graph which showed change of maximum energy product (BH) max to Ga content of a Nd-Fe-B mold sintered magnet, coercive force iHc, and corrosion weight loss.
[Drawing 5] Maximum energy product (BH) max to Ga content (0 - 0.6wt%) and Dy content of a Nd-Fe-B mold sintered magnet, the graph which showed change of coercive force iHc.
[Drawing 6] The graph which showed change of maximum energy product (BH) max to Ga content (0 - 0.6wt%) of a Nd-Fe-B mold sintered magnet, coercive force iHc, and square shape nature.
[Drawing 7] The sintered compact mean particle diameter to Nb content of a Nd-Fe-B mold sintered magnet, the graph which showed change of maximum energy product (BH) max.

[Translation done.]

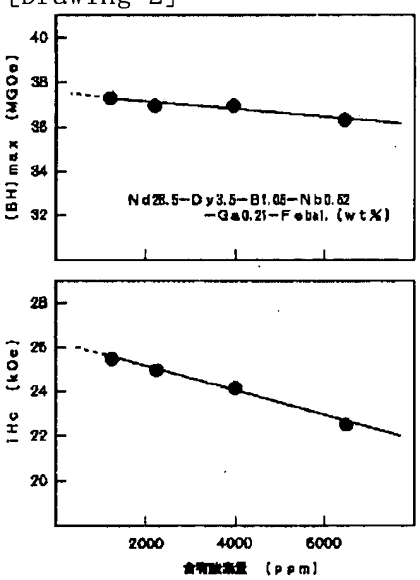
2. **** shows the word which can not be translated.
3. In the drawings, any words are not translated.

DRAWINGS

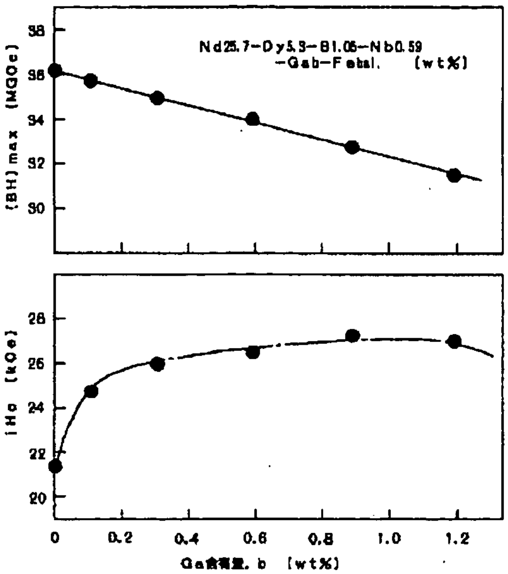
[Drawing 1]



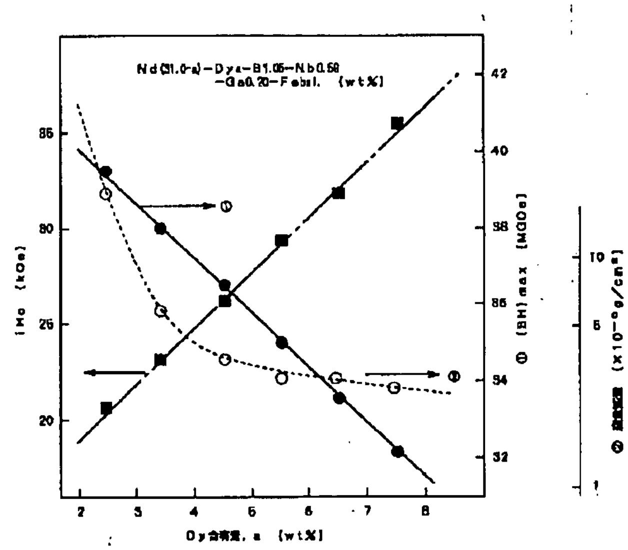
[Drawing 2]



[Drawing 3]



[Drawing 4]



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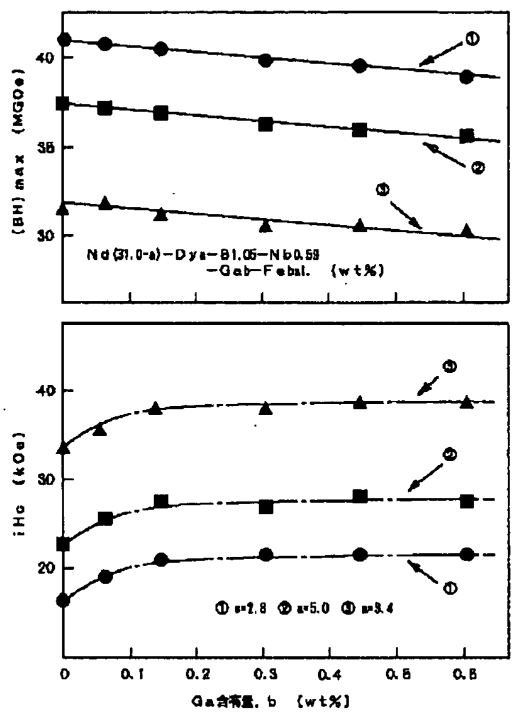

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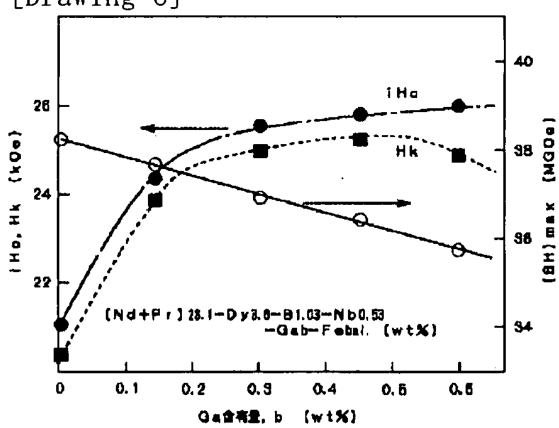
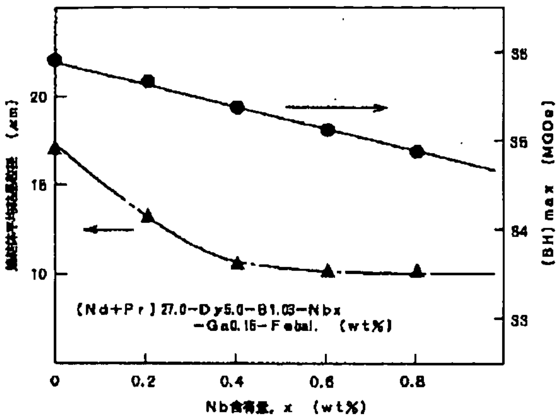


[Drawing 7]



[Translation done.]